20 mL, 100 mm). Prepare the solution of Ergocalciferol within 30 minutes after the container has been opened, and determine the rotation within 30 minutes after the solution has been prepared.

Purity Ergosterol—Dissolve 0.010 g of Ergocalciferol in 2.0 mL of diluted ethanol (95) (9 in 10), add a solution of 0.020 g of digitonin in 2.0 mL of diluted ethanol (95) (9 in 10), and allow the mixture to stand for 18 hours: no precipitate is formed.

Assay Weigh accurately about 0.03 g each of Ergocalciferol and Ergocalciferol Reference Standard, and dissolve each in isooctane to make exactly 50 mL. Pipet 10 mL each of these solutions, add exactly 3 mL each of the internal standard solution, then add the mobile phase to make 50 mL, and use these solutions as the sample solution and the standard solution, respectively. Perform the test with 10 to 20 μL each of the sample solution and the standard solution as directed under the Liquid Chromatography according to the following conditions, and calculate the ratios, Q_T and Q_S, of the peak area of ergocalciferol to that of the internal standard. Perform the procedure rapidly avoiding contact with air or other oxidizing agents and using light-resistant containers.

$$\text{Amount (mg) of } C_{28}H_{44}O = \text{amount (mg) of Ergocalciferol Reference Standard} \times \frac{Q_T}{Q_S}$$

Internal standard solution—A solution of dimethyl phthalate in isooctane (1 in 100).

Operating conditions—

Detector: An ultraviolet absorption photometer (wavelength: 254 nm).

Column: A stainless steel column about 4 mm in inside diameter and 10 to 30 cm in length, packed with a silica gel for liquid chromatography (5 to 10 μm particle diameter).

Column temperature: Ordinary temperature.

Mobile phase: A mixture of hexane and *n*-amylalcohol (997:3).

Flow rate: Adjust the flow rate so that the retention time of ergocalciferol is about 25 minutes.

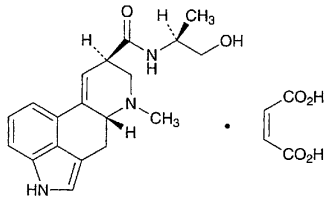
Selection of column: Dissolve 0.015 g of Ergocalciferol Reference Standard in 25 mL of isooctane. Transfer this solution to a flask, heat in an oil bath under a reflux condenser for 2 hours, and cool immediately to room temperature. Transfer the solution to a quartz test tube, and irradiate with a short-wave lamp (main wavelength: 254 nm) and a long-wave lamp (main wavelength: 365 nm) for 3 hours. To 10 mL of this solution add the mobile phase to make 50 mL. Proceed with 10 μL of this solution under the above operating conditions. Use a column with the ratios of the retention time of previtamin D_2, trans-vitamin D_2 and tachysterol$_2$ to that of ergocalciferol being about 0.5, about 0.6 and about 1.1, respectively, and with resolution between previtamin D_2 and trans-vitamin D_2 being not less than 0.7, and that between ergocalciferol and tachysterol$_2$ being not less than 1.0.

Containers and storage Containers—Hermetic containers.

Storage—Light-resistant, under Nitrogen atmosphere, and in a cold place.

Ergometrine Maleate

マレイン酸エルゴメトリン



$C_{19}H_{23}N_3O_2.C_4H_4O_4$: 441.48

(8*S*)-9,10-Didehydro-*N*-[(1*S*)-2-hydroxy-1-methylethyl]-6-methylergoline-8-carboxamide monomaleate [*129-51-1*]

Ergometrine Maleate, when dried, contains not less than 98.0% of $C_{19}H_{23}N_3O_2.C_4H_4O_4$.

Description Ergometrine Maleate occurs as a white to pale yellow, crystalline powder. It is odorless.

It is sparingly soluble in water, slightly soluble in methanol and in ethanol (95), and practically insoluble in diethyl ether.

Melting point: about 185°C (with decomposition).

It gradually changes to yellow in color on exposure to light.

Identification **(1)** Prepare a solution of Ergometrine Maleate (1 in 50): the solution shows a blue fluorescence.

(2) Dissolve 1 mg of Ergometrine Maleate in 5 mL of water. To 1 mL of this solution add 2 mL of 4-dimethylaminobenzaldehyde-ferric chloride TS, shake, and allow to stand for 5 to 10 minutes: a deep blue color develops.

(3) To 5 mL of a solution of Ergometrine Maleate (1 in 500) add 1 drop of potassium permanganate TS: the red color of the solution disappears immediately.

Optical rotation $[\alpha]_D^{20}$ +48 – +57° (after drying 0.25 g, water, 25 mL, 100 mm).

pH Dissolve 0.10 g of Ergometrine Maleate in 10 mL of water. The pH of the solution is between 3.0 and 5.0.

Purity **(1)** Clarity and color of solution—Dissolve 0.10 g of Ergometrine Maleate in 10 mL of water: the solution is clear and colorless to light yellow.

(2) Ergotamine and ergotoxine—To 0.02 g of Ergometrine Maleate add 2 mL of a solution of sodium hydroxide (1 in 10), and heat to boiling: the gas evolved does not change moistened red litmus paper to blue.

(3) Related substances—Dissolve 5 mg each of Ergometrine Maleate and Ergometrine Maleate Reference Standard in 1.0 mL of methanol, and use these solutions as the sample solution and the standard solution, respectively. Perform the test with these solutions as directed under the Thin-layer Chromatography. Spot 10 μL each of the sample solution and the standard solution on a plate, prepared with silica gel for thin-layer chromatography and dilute sodium hydroxide TS. Develop the plate with a mixture of chloroform and methanol (4:1) to a distance of about 10 cm, and air-dry the plate. Spray evenly 4-dimethylaminobenzaldehyde TS on the plate: the spots obtained from the sample solution and the standard solution show a red-purple color

and the same *Rf* value, and any spot from the sample solution other than that corresponding to the spot from the standard solution does not appear.

Loss on drying Not more than 2.0% (0.2 g, silica gel, 4 hours).

Assay Weigh accurately about 0.01 g each of Ergometrine Maleate and Ergometrine Maleate Reference Standard, previously dried in a desiccator (silica gel) for 4 hours, dissolve in water to make exactly 250 mL, and use these solutions as the sample solution and the standard solution, respectively. Pipet 2 mL of each solution into a separate brown glass-stoppered tube. To each tube add 4 mL of 4-dimethylaminobenzaldehyde-ferric chloride TS, exactly measured, while cooling in an ice bath, then warm at 45°C for 10 minutes. Allow to stand at room temperature for 20 minutes, and perform the test with these solutions as directed under the Ultraviolet-visible Spectrophotometry, using a solution, prepared with 2 mL of water in the same manner, as the blank. Determine the absorbances, A_T and A_S, of the subsequent solutions of the sample solution and the standard solution at 550 nm, respectively.

Amount (mg) of $C_{19}H_{23}N_3O_2.C_4H_4O_4$
= amount (mg) of Ergometrine Maleate Reference Standard
$\times \frac{A_T}{A_S}$

Containers and storage Containers—Tight containers.
Storage—Light-resistant.

Assay Transfer an exactly measured volume of Ergometrine Maleate Injection, equivalent to about 2 mg of ergometrine maleate ($C_{19}H_{23}N_3O_2.C_4H_4O_4$), and add sodium chloride in a ratio of 0.3 g to 1 mL of the solution. To this mixture add 20 mL of diethyl ether and 2 mL of ammonia TS, shake, and extract. Further, extract with three 15-mL portions of diethyl ether, combine all the extracts, add 5 g of anhydrous sodium sulfate, filter through a pledget of absorbent cotton, and wash with three 5-mL portions of diethyl ether. Add the washings to the filtrate, shake with 5 mL of dilute sulfuric acid, evaporate the diethyl ether by warming in a current of nitrogen, to the remaining solution add water to make exactly 50 mL, and use this solution as the sample solution. Weigh accurately about 2 mg of Ergometrine Maleate Reference Standard, previously dried in a desiccator (silica gel) for 4 hours, add water to make exactly 50 mL, and use this solution as the standard solution. Transfer 2 mL each of the sample solution and the standard solution, accurately measured, to separate glass-stoppered test tubes, and proceed as directed in the Assay under Ergometrine Maleate.

Amount (mg) of ergometrine maleate ($C_{19}H_{23}N_3O_2.C_4H_4O_4$)
= amount (mg) of Ergometrine Maleate Reference Standard
$\times \frac{A_T}{A_S}$

Containers and storage Containers—Hermetic containers, and colored containers may be used.
Storage—Light-resistant, and in a cold place.

Ergometrine Maleate Injection

マレイン酸エルゴメトリン注射液

Ergometrine Maleate Injection is an aqueous solution for injection. It contains not less than 90% and not more than 110% of the labeled amount of ergometrine maleate ($C_{19}H_{23}N_3O_2.C_4H_4O_4$: 441.48).

Method of preparation Prepare as directed under Injections, with Ergometrine Maleate.

Description Ergometrine Maleate Injection is a clear, colorless to pale yellow liquid.
pH: 2.7 - 3.5

Identification **(1)** Measure a volume of Ergometrine Maleate Injection, equivalent to 3 mg of Ergometrine Maleate according to the labeled amount, if necessary, dilute with water or evaporate on a water bath to make 15 mL, and use this solution as the sample solution. The sample solution shows a blue fluorescence.

(2) To 1 mL of the sample solution obtained in (1) add 1 mL of ammonia TS, and extract with 20 mL of diethyl ether. To the diethyl ether extract add 1 mL of dilute sulfuric acid, shake, and warm to remove diethyl ether in a water bath. Cool, to the residue obtained add 2 mL of 4-dimethylaminobenzaldehyde-ferric chloride TS, and allow to stand for 5 to 10 minutes: a deep blue color develops.

(3) To 5 mL of the sample solution obtained in (1) add 1 drop of potassium permanganate TS: a red color disappears immediately.

Ergometrine Maleate Tablets

マレイン酸エルゴメトリン錠

Ergometrine Maleate Tablets contain not less than 90% and not more than 110% of the labeled amount of ergometrine maleate ($C_{19}H_{23}N_3O_2.C_4H_4O_4$: 441.48).

Method of preparation Prepare as directed under Tablets, with Ergometrine Maleate.

Identification To a quantity of powdered Ergometrine Maleate Tablets, equivalent to 3 mg of Ergometrine Maleate according to the labeled amount, add 15 mL of warm water, shake, and filter: the filtrate shows a blue fluorescence. Proceed with this solution as directed in the Identification (2) and (3) under Ergometrine Maleate.

Content uniformity Transfer 1 tablet of Ergometrine Maleate Tablets to a glass-stoppered centrifuge tube, and add a solution of L-tartaric acid (1 in 100) to make exactly *V* mL of a solution containing about 0.04 mg of ergometrine maleate ($C_{19}H_{23}N_3O_2.C_4H_4O_4$) per ml. Stopper the tube, shake for 30 minutes vigorously, centrifuge, and use the supernatant liquid as the sample solution. Separately, weigh accurately about 4 mg of Ergometrine Maleate Reference Standard, previously dried in a desiccator (silica gel) for 4 hours, dissolve in water to make exactly 100 mL, and use this solution as the standard solution. Pipet 4 mL each of the sample solution and the standard solution into separate brown glass-stoppered test tubes, add exactly 8 mL each of 4-